

## TRANSLATION

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### (54) Title of the Invention: Method to Produce the High Strength Porous Ceramics

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### D scription

1. Titl of th Invention : M thod to Produce the High Strength Porous C ramics

## **2. Scope of the Patent Application**

(1) Method to produce the porous ceramics with high strength characterized by the fact that it uses the needle- like mullite powder that can be obtained when a clay mineral, of which the main components are silicon dioxide and aluminum oxide, is baked and then the co-existing glass component is eluted out with an acid or an alkali, and that after it is formed, the ceramic is constructed by only the needle- like mullite.

(2) Method to produce the porous ceramics with high strength characterized by the fact that its pore properties can be controlled freely by forming and baking after mixing the above mentioned needle- like mullite powder with an organic material that will disappear by combustion.

## **3. Detailed Explanation of the Invention**

### **[Field of Utilization in Industry]**

This invention relates to the method to produce the new porous ceramics with high strength which can be used for the various catalyst supports under oxidizing atmospheres at 1000 to 1700 °C or as the gas filter to remove the fine particles from the gas, and for the precision filtration of liquid metals.

### **[Existing Technology]**

Concerning the existing methods to produce the porous ceramics, there are the following methods:

(1) the method in which the ceramic powder of which the grain size was controlled is formed by using an inorganic or organic binder, and thereafter it is baked.

(2) the method in which the polymer powder, organic fiber, carbon powder, etc., are mixed into the ceramic powder as the pore forming material, and it is formed and baked

to remove the combustible material completely, and thus, the pores remain in the ceramic.

(3) the method in which ceramic slip is impregnated with polymer foam material, and thereafter, this polymer foam material is removed by heat treatment.

**[Problem That this Invention Intends to Solve]**

In the porous ceramics produced by the existing methods, the glass type flux or clay type materials are used as a binder to bind each of the ceramic particles, so that the heat resistance or chemical resistance of the porous body tends to decrease. The porous body has generally low strength (flexural strength), and the strength values reported until now are mostly around 200 kgf / cm<sup>2</sup>.

In addition, under the high temperature conditions, the porosity or the pore diameters tend to change easily due to the binder becoming soft, and in the case when the bonding between the particles is weak, the strength decreases greatly, and these were the problems.

**[Method to Solve the Problem]**

The inventors of this invention have made the following clear until now; that the needle-like or whisker-like mullite that is baked and extracted from the clay mineral:

- (1) is low in the content of alkali impurities which tend to easily form a glass phase,
- (2) has heat resistance to about 1700 °C in air and its shape does not change easily, and
- (3) it has a relatively low coefficient of thermal expansion.

(Nihon Ceramics Kyokai Gakujutsu Ronbunshi (Japan Ceramic Association Academic Journal Vol. 97 (1989))

They paid attention to the fact that when this crystal intermingles in three dimensions by sintering, the continuous space can be formed internally, and they discovered that the ceramics with high strength which have excellent porous body properties (such as porosity or pore diameter, etc.) at high temperature, can be produced by a simple process in which it is formed into the desired form by only needle- like mullite and then it is baked. Namely, only the needle- like mullite of which the length is 1 to 20  $\mu\text{m}$  and the width is 0.1 to 3  $\mu\text{m}$ , is formed into plates, pipes or honey comb shapes by the mold press forming, cast molding, or extrusion forming, etc., and then it is baked in air at 1500 to 1700  $^{\circ}\text{C}$ . ; or the combustible material, for example a polymer powder such as acrylic resin, poly ethylene, etc., or activated carbon, carbon fiber powder, are mixed into the needle- like mullite, and it is formed and the combustible material is removed in air, and thus, the porous ceramics of which the porous body properties (porosity and pore distribution) are controlled freely, and also which have high strength, can be obtained. The Al component or the Si component in mullite mutually disperse on each crystal surface at 1500 to 1700  $^{\circ}\text{C}$ , so that the sintering between the crystals tends to occur easily. Therefore, the binding assist agents or sintering assist agents which used to be necessary with the existing methods to produce the porous body to bond the constructing particles, becomes totally unnecessary in the method of this invention. In addition, mullite is an oxide with strong covalent bonding ability, therefore, once the bond is formed between the crystals, the bonding interface becomes strong, so that the strength of the porous body constructed with the needle- like mullite increases. Also, the porosity or pore diameter can be controlled freely by the size or the amount of the combustible material that is added.

**[Actual Examples]**

Next, actual examples of this invention will be explained.

**Example 1**

About 30 g of needle- like mullite powder was put into a metal mold with a 60 mm diameter, and it was formed (primary forming) at  $200 \text{ kgf / cm}^2$ . Thereafter, it was formed by hydrostatic pressing at 1 to 4 ton /  $\text{cm}^2$ , and it was baked in air at 1500 to 1700 °C for 1 to 5 hours. The average of three measured points for flexural strength, porosity, pore diameter, and the average thermal expansion coefficient from room temperature to 1400 °C, of each of the porous bodies obtained under each condition, are shown in Figure 1. Especially, the properties of the porous bodies that were baked at 1650 °C or higher, almost do not change at all, even if they are re-heated at 1000 to 1650 °C for several tens of hours in air.

**Example 2**

1 to 2 g of binder, 3 to 4 g of peptizer, and 12 to 13 g of pure water were added into 50 g of needle- like mullite, and it was sufficiently mixed. This slurry was formed by casting into a gypsum mold, and the plate - like formed body of which the length was 50 mm, the thickness was about 5 mm, and the width was 30 mm, was obtained. After it was dried, it was baked in air at 1500 to 1650 °C for 1 to 5 hours. Figure 2 shows the average of three measured points of flexural strength, porosity, pore diameter, and the average thermal expansion coefficient from room temperature to 1400 °C, for each porous body obtained under each set of conditions. Needle- like mullite becomes slightly more easily oriented to the gypsum surface, so that the strength increases, compared with the porous body obtained with a metal mold.

**Example 3**

10 g of carbon fiber powder, of which the diameter was 10  $\mu\text{m}$  and the length was 100 to 200  $\mu\text{m}$ , 2 g of methyl cellulose, and 40 g of pure water were added into 20 g of needle- like mullite, and it was sufficiently mixed. Then, it was formed by casting into a plate (5 x 20 x 50 mm). After it was dried, it was baked at 1600 to 1700 °C for 2 hours.

At 1600 °C, the porous body of which the average of three measured points of flexural strength was 350 to 400 kgf / cm<sup>2</sup>, and the porosity was 49 to 52 %, could be obtained, however, the pore diameter was distributed sharply at 0.7 ~ 0.8 μm and 6 ~ 7 μm. This indicates the distribution of the pores formed by needle- like mullite, and the pores formed by disappearance of carbon fibers by combustion. Also at 1700 °C, the porous body of which the average of three measured points of flexural strength was 480 to 530 kgf / cm<sup>2</sup>, and the porosity was 36 to 41 %, could be obtained, however, the pore diameters are distributed at 0.4 ~ 0.6 μm and 4 ~ 5 μm.

Thus, the pore properties of the porous bodies made out of needle- like mullite can be controlled by adding a combustible material (the size, amount added, etc., can be adjusted).

#### 4. Simple Explanation of Figures

**Figure 1** is the table which shows the results of determining various properties of the porous body obtained by baking it after needle- like mullite was formed by hydrostatic pressing.

**Figure 2** is the table which shows the results of determining various properties of the porous body obtained by baking it after cast forming.

**Figure 3** is the results of determining the pore distribution of the porous body obtained by adding carbon fiber powder into needle- like mullite and thereafter by cast forming and baking it.

*[Some numbers in the copy of the table used for translation were not clear, please refer to the original, Translator]*

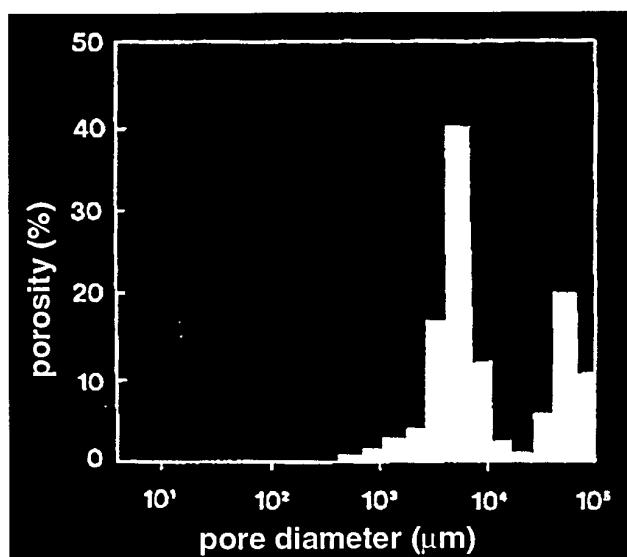
**Figure 1**

Hydrostatic pressure (ton / cm <sup>2</sup> )	Baking temperature (°C)	Flexural strength (kgf / cm <sup>2</sup> )	Porosity (%)	Pore diameter (μm)	Thermal expansion coefficient (x 10 <sup>-4</sup> / °C)
0	1600	380?	57.8	0.66	4.?
1	1600	710	55.?	0.78	4.5
2	1600	780?	49.9	0.43	4.7
3	1600	800	42.5	0.39	4.7
4	1600	850	37.6	0.35	5.0
2	1500	450	58.5?	0.65	4.4
0	1550	280?	60.2	0.77	4.?
2	1550	540	48.7	0.40	4.7
0	1650	455	37.6	0.62	4.9
2	1650	865	31.5	0.36	5.1
2	1700	980?	24.5	0.??	5.5

**Figure 2**

Baking temperature (°C)	Flexural strength (kgf / cm <sup>2</sup> )	Porosity (%)	Pore diameter (μm)	Thermal expansion coefficient (x 10 <sup>-6</sup> / °C)
1500	485	58.1	0.75	4.4
1550	580	52.5	0.66	4.8
1600	790	42.1	0.62	4.5
1650	1050	29.4	0.58	4.9

Figure 3



*[Pages 4, 5, 6 and 7 were somewhat complicated correction pages. All these corrections were made before starting translation, Translator.]*